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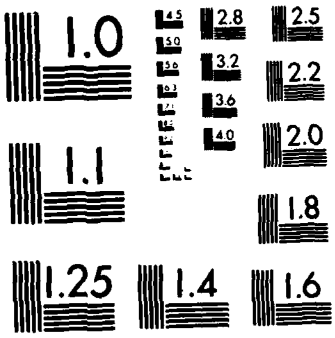

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by

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DEFENCE RESEARCH ESTABLISHMENT OTTAWA
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ABSTRACT

The Setapoint apparatus, and a procedure originally developed by British Petroleum, are designed to give a measure of the minimum temperature at which middle distillate fuels can be used. These were investigated, employing an experimental fuel, ERBS, of petroleum origin, and synthetic mixtures based on isooctane with added n-paraffins. Special attention was given to the effects of cold flow improvers.

RÉSUMÉ

L'appareil Setapoint et une méthode mise au point par la British Petroleum ont été conçus en vue de mesurer la température minimale d'utilisation de carburants constitués d'un distillat moyen. Cet appareil et cette méthode ont été étudiés en utilisant un carburant expérimental, le ERBS, qui est dérivé du pétrole, et des mélanges synthétiques basés sur l'iso-octane additionné de n-paraffines. On a étudié de façon particulière l'effet des agents améliorant l'écoulement à froid.

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1.0.0 INTRODUCTION

In previous work (1) we compared a number of methods for determining the low temperature flow behaviour of aviation gas turbine fuels. The materials used included both specification fuels and variants of higher, sometimes considerably higher, freeze point. Interest in this subject arose from the recognition that the use of higher freezing fuels might become necessary in the future, as one option in a straitened supply situation.

Two bench tests, the Setapoint and a measure of flow resistance in a capillary, developed by British Petroleum, were found to correlate well with two larger scale tests which simulate aspects of flow behaviour in aircraft fuel tanks. These latter tests require considerable time to produce a single data point, as it is necessary to cool down the apparatus containing fuel repetitively from room temperature to a succession of decreasing temperatures; while the Seta and BP methods require small samples, and a series of determinations can be quickly run. The work to be described here is a comparison of these two test methods.

The use of cold flow improvers, or pour depressants, with heavier aircraft fuels has not received much attention. In the previous report covering a considerable variety of fuels and test methods, we briefly examined two flow improvers. In the present work we compared the two test methods (Seta and capillary flow) in an examination of a wide array of these materials, with a high freeze point fuel. For this purpose ERBS (Experimental Referee Broadened Specification) fuel appeared the most suitable to begin with. ERBS has been promoted by NASA as a reference fuel for research purposes, and, although based on NASA's perception of the American supply situation in the 1970's, is representative of what a high density, high freeze point fuel would look like, if resort to such a fuel became necessary. The divergence in its properties from existing fuel specifications is, in some respects, extreme; the freeze point, for example, about -25°C, is unacceptably high.

Low temperature usability of fuels is determined in the first instance by the wax (n-paraffin) composition, since n-paraffins deposit as a class at higher temperatures than other compound types in the same molecular weight range. Flow improvers act primarily by modifying the physical structure of deposited wax, but presumably their action is affected by other fuel constituents. As an introduction to some projected work on the role of fuel composition we carried out a similar survey of flow improvers using as fuel isooctane to which waxes had been added to reproduce quantitatively the wax distribution found in ERBS. A limited amount of testing was carried out also with isooctane containing an untypical wax distribution, peaking in the C_{15} - C_{16} range.

TABLE I

FLOW IMPROVERS AND OTHER ADDITIVES

COMPOUND	SOURCE
PARADYNE 25	ESSO CHEMICALS CANADA
PARAMINS ECA 7973	ESSO CHEMICALS CANADA
HITEC E-623	ETHYL CANADA INC.
HITEC E-672	ETHYL CANADA INC.
LUBRIZOL 8202	LUBRIZOL OF CANADA LTD.
LUBRIZOL 8052	LUBRIZOL OF CANADA LTD.
TOLAD T-31	PETROLITE CORPORATION OF CANADA LTD.
TOLAD T-35	PETROLITE CORPORATION OF CANADA LTD.
TOLAD T-40	PETROLITE CORPORATION OF CANADA LTD.
TOLAD T-346	PETROLITE CORPORATION OF CANADA LTD.
TOLAD T-2121	PETROLITE CORPORATION OF CANADA LTD.
CHEVRON XF-3	CHEVRON RESEARCH LABORATORY
POLYVINYL PYRROLIDONE NP-K15	GAF (CANADA) INC.
ANTAROX CO 430	GAF (CANADA) INC.
ANTAROX CO 530	GAF (CANADA) INC.
ANTAROX DM 430	GAF (CANADA) INC.
ANTAROX DM 530	GAF (CANADA) INC.
ANTARON V 216	GAF (CANADA) INC.
GANEX V 220	GAF (CANADA) INC.
GANTREZ ES 425	GAF (CANADA) INC.
ACRYLOID 150	ROHM AND HAAS CANADA LTD.
ACRYLOID 144-50	ROHM AND HAAS CANADA LTD.
ACRYLOID 154-70	ROHM AND HAAS CANADA LTD.

2.0.0 EXPERIMENTAL

2.1.0 APPARATUS

The Setapoint apparatus has already been described (1). Its principle of operation is a measurement of the resistance to flow as a fuel, under programmed cooling is forced back and forth across a metal screen. The capillary flow test depends upon the resistance of a long fuel column to displacement induced by air pulses applied at 1°C intervals while the fluid cools. Two types of behaviour were observed in the previous report - a very rapid freeze up, so that the fuel passes from fluid to completely immobile within a 1°C interval, and gradual increase in viscosity and resistance to motion, necessitating the adoption of some arbitrary criterion for the end point.

2.1.1 Materials

ERBS-3 is one of several variations in this reference fuel, that have been characterized extensively by Seng (2). The additives used here are listed in Table I. They include both recognized flow improvers or pour depressants for middle distillates and crudes, and a number of materials of surfactant type, some of them not obvious candidates in this application, but thought to be worth screening in view of the paucity of information on the subject. Some are supplied as pure or nearly pure compounds, others in a hydrocarbon base of varying, sometimes unstated concentration. For consistency we expressed all concentrations as weight per cent of the material as supplied.

Isooctane (2,2,4 trimethylpentane), a highly branched compound of very low freeze point, was adopted as a suitable indifferent base to which the straight chain waxes were added. In preparing isooctane with the ERBS-3 distribution of waxes, the analytical results of Seng (2) were employed:

C_8 - 0.3, C_9 - 0.6, C_{10} - 2.0, C_{11} - 4.7, C_{12} - 6.4, C_{13} - 5.0, C_{14} - 2.2, C_{15} - 1.1, C_{16} - 0.9, C_{17} - 0.8, C_{18} - 0.5, C_{19} - 0.3, C_{20} - 0.1, all as weight per cent of the fuel.

The effect of flow improvers was also examined, using a 10% w/w solution in isooctane of the eight n-paraffins from C_{12} to C_{19}, in the amounts:

C_{12} - 0.38, C_{13} - 0.77, C_{14} - 1.53, C_{15} - 2.31, C_{16} - 2.31, C_{17} - 1.53, C_{18} - 0.77, C_{19} - 0.38 wt% i.e., in the relative ratios 1:2:4:6:6:4:2:1.

3.0.0 RESULTS

Setapoint determinations on ERBS-3, alone and with the 23 candidate flow improvers at several levels, are summarized in Table II. The results with flow improvers at the 0.1% level are depicted in Fig. 1. With ERBS-3 there is a considerable spread between stop flow (-31.5°C) and resume flow (-25.8°C), due to supercooling. In presenting the results in Fig. 1, all stop and resume flow points with the additives have been plotted as relative to the corresponding values for the untreated ERBS. Thus, the bars in Fig. 1 are a measure of the improvement (and the occasional deterioration) in performance effected by the additive.

The most effective additives were the Tolads (Except Tolad T-40), the Lubrizols, Ganex V 220, Paradyne 25 and Paramins ECA 7973. The Antarox series was totally ineffective, as were Acryloid 144-50, Gantrez ES-425 and PVP NP-K15. These last two were only slightly soluble in fuel, and tests were conducted with saturated solutions of these materials, of concentrations roughly 0.01%

Corresponding results with isooctane containing the ERBS-3 distribution of waxes are listed in Table III and plotted, in the same way as before, in Fig. 2. The additives that had been found completely ineffective with ERBS itself were eliminated from consideration, and do not appear. To facilitate comparison, each additive occupies the same position in all the figures in this note.

Comparing Figures 1 and 2, one observes:

a) the profile of the bar charts are similar, indicating that results for the series of additives on the two systems parallel each other. Flow improvers that are effective with ERBS are in nearly every case correspondingly effective with the synthetic blend; thus the wax composition is the major determinant of flow improver action.

b) the magnitude of Setapoint depressions with ERBS is considerably greater than with the isooctane-based fuel.

c) there are several obvious discrepancies, the reversal in the order of effectiveness of Ganex V 220 and Antaron V 216 in the two systems, and the greatly diminished effectiveness of Lubrizol 8202 and Hitec E672 in the isooctane blend.

These last two observations show that fuel composition factors other than wax content can influence flow improver action.

The effects of additives on the same two systems, ERBS and isooctane with the ERBS distribution of waxes, were next investigated using changes in the capillary stop flow temperature as a measure of their effectiveness. Results for ERBS are presented in Table IV and plotted in Figure 3, and for the synthetic blend in Table V.

TABLE II

SETAPOINT RESULTS FOR ERBS-3

ADDITIVE	%W/W CONC	SETA STOP FLOW	SETA RESUME FLOW	ADDITIVE	%W/W CONC	SETA STOP FLOW	SETA RESUME FLOW
(NONE)	-	-31.5	-25.8	ANTAROX CO 430	0.1	-29.4	-23.8
ACRYLOID 150	0.1	-31.3	-23.3	ANTAROX CO 530	0.1	-29.4	-24.0
ACRYLOID 150	0.2	-40.4	-34.8	ANTAROX CO 530	0.2	-29.1	-24.4
ACRYLOID 144-50	0.1	-32.3	-28.7	ANTAROX DM 430	0.1	-32.4	-28.7
ACRYLOID 144-50	0.2	-30.8	-27.8	ANTAROX DM 530	0.1	-31.3	-28.4
ACRYLOID 154-70	0.1	-38.8	-29.6	ANTARON V 216	0.1	-34.8	-28.9
ACRYLOID 154-70	0.2	-43.7	-36.8	ANTARON V 216	0.2	-43.5	-34.9
TOLAD T-31	0.1	-42.5	-42.6	GANEX V 220	0.05	-44.5	-44.0
TOLAD T-35	0.1	-44.2	-41.8	GANEX V 220	0.1	-45.2	-44.5
TOLAD T-40	0.1	-37.2	-32.0	GANEX V 220	0.2	-43.8	-43.2
TOLAD T-346	0.1	-47.2	-40.2	GANTREZ ES 425	SAT'D	-31.5	-27.2
TOLAD T-2121	0.05	-44.9	-44.3	PVP NP K15	SAT'D	-29.5	-23.9
TOLAD T-2121	0.1	-46.1	-45.1	PARADYNE 25	0.05	-48.3	-44.8
TOLAD T-2121	0.2	-47.8	-46.0	PARADYNE 25	0.1	-48.2	-45.1
HITEC E-623	0.1	-34.1	-29.1	PARAMINS ECA7973	0.05	-45.5	-43.0
HITEC E-623	0.2	-39.6	-31.0	PARAMINS ECA7973	0.1	-47.6	-44.8
HITEC E-672	0.1	-39.3	-26.6	PARAMINS ECA7973	0.2	-47.9	-45.7
LUBRIZOL 8052	0.05	-47.8	-43.8				
LUBRIZOL 8052	0.1	-47.5	-44.1				
LUBRIZOL 8202	0.05	-43.7	-43.6				
LUBRIZOL 8202	0.1	-43.7	-43.1				
CHEVRON XF-3	0.1	-38.7	-33.2				
CHEVRON XF-3	0.2	-36.3	-35.6				

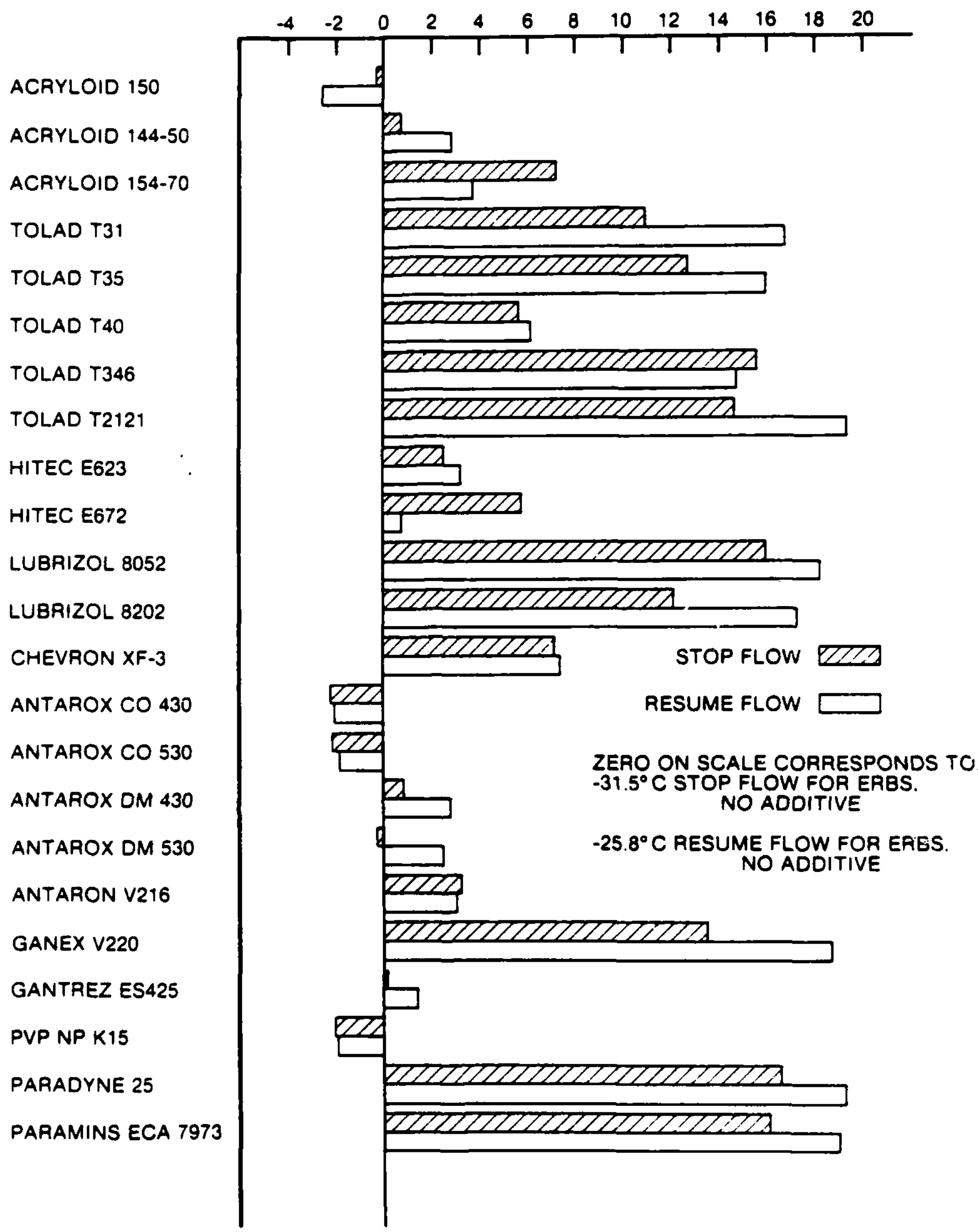

FIGURE 1. EFFECT OF 23 FLOW IMPROVERS ON SETAPOINT STOP AND FLOW POINTS OF ERBS-3

TABLE III

SETAPOINT RESULTS FOR ISOOCTANE + ERBS-3
WAX DISTRIBUTION

ADDITIVE	%W/W CONC	SETA STOP FLOW	SETA RESUME FLOW	ADDITIVE	%W/W CONC	SETA STOP FLOW	SETA RESUME FLOW
(NONE)	-	-34.7	-34.1	HITEC E 672	0.1	-36.9	-33.0
ACRYLOID 150	0.1	-35.5	-29.1	LUBRIZOL 8202	0.1	-39.5	-37.6
ACRYLOID 154-70	0.1	-38.7	-34.3	LUBRIZOL 8052	0.1	-45.6	-43.8
TOLAD T-31	0.1	-39.7	-37.6	CHEVRON XF-3	0.1	-44.5	-41.7
TOLAD T-35	0.1	-39.9	-40.0	ANTARON V 216	0.1	-42.8	-41.3
TOLAD T-40	0.1	-37.2	-36.8	GANEX V 220	0.1	-40.3	-40.3
TOLAD T-346	0.1	-38.1	-37.7	PARADYNE 25	0.1	-45.8	-43.5
TOLAD T-2121	0.1	-43.9	-43.0	PARAMINS ECA7973	0.1	-44.0	-42.8
HITEC E-623	0.1	-38.4	-36.3				

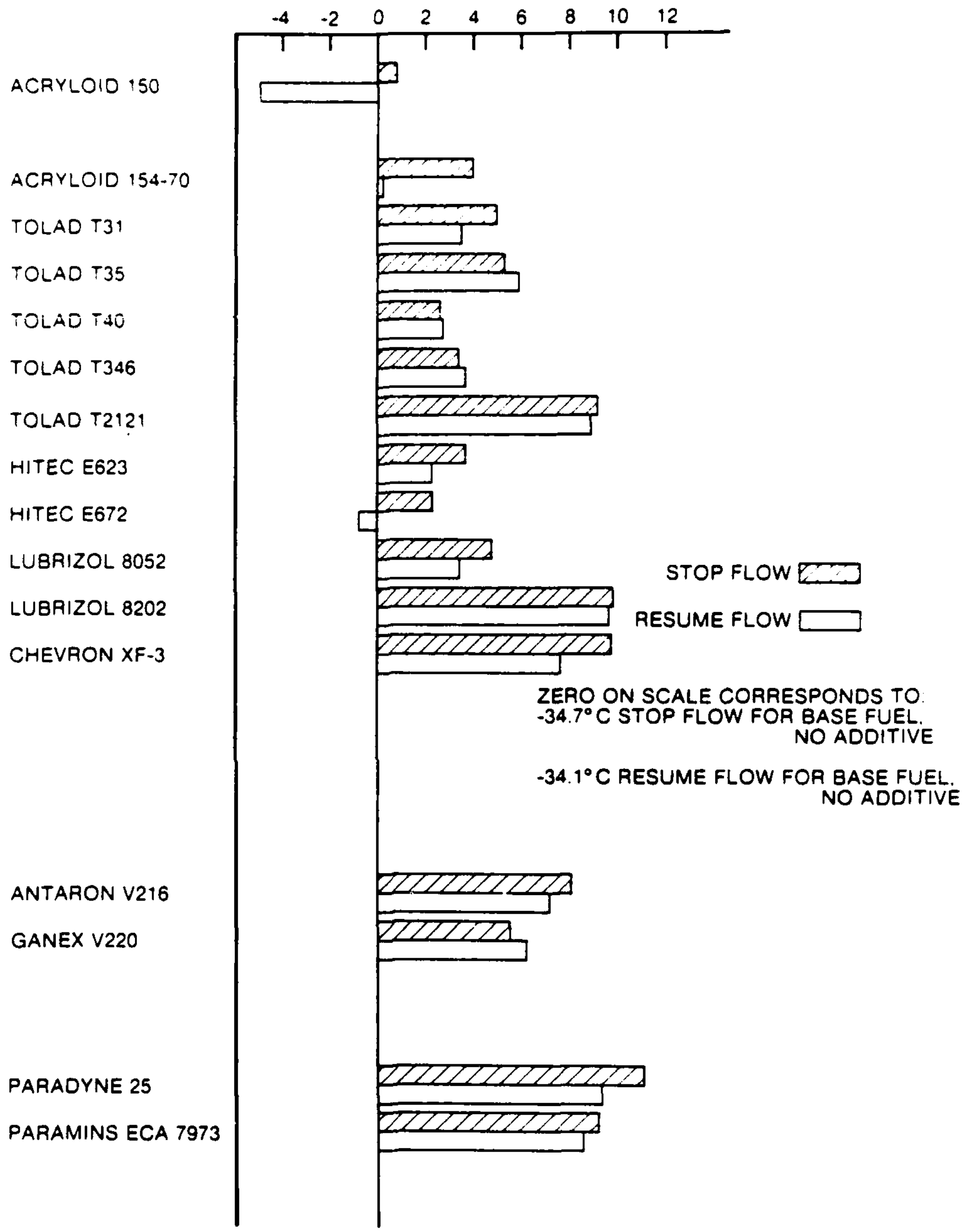

FIGURE 2. EFFECT OF 16 FLOW IMPROVERS ON CAPILLARY TEST RESULTS FOR ERBS-3

TABLE 1V

CAPILLARY TEST RESULTS FOR ERBS-3

ADDITIVE	%W/W CONC	STOP FLOW	ADDITIVE	%W/W CONC	STOP FLOW
(NONE)	-	-42	CHEVRON XF-3	0.1	-49
ACRYLOID 150	0.1	-50	ANTAROX CO 430	0.1	-43
ACRYLOID 150	0.2	-50.5	ANTAROX CO 530	0.1	-43
ACRYLOID 144-50	0.1	-47	ANTAROX CO 530	0.2	-40
ACRYLOID 144-50	0.2	-48	ANTAROX DM 430	0.1	-43
ACRYLOID 154-70	0.05	-50	ANTAROX DM 530	0.1	-41
ACRYLOID 154-70	0.1	-52	ANTARON V 216	0.05	-50
TOLAD T-31	0.1	-47	ANTARON V 216	0.1	-51
TOLAD T-31	0.2	-47	ANTARON V 216	0.2	-50
TOLAD T-35	0.1	-50	GANEX V 220	0.05	-45
TOLAD T 40	0.1	-50	GANEX V 220	0.1	-46
TOLAD T-40	0.2	-50	GANEX V 220	0.2	-46.5
TOLAD T-346	0.1	-50	GANTREZ ES 425	SAT'D	-44
TOLAD T-2121	0.05	-49	PVP NP K15	SAT'D	-43
TOLAD T-2121	0.1	-50	PARADYNE 25	0.05	-50
TOLAD T-2121	0.2	-50	PARADYNE 25	0.1	-51
HITEC E-623	0.1	-52	PARAMINS ECA7973	0.05	-49
HITEC E-623	0.2	-52	PARAMINS ECA7973	0.1	-49
HITEC E-672	0.1	-50.5	PARAMINS ECA7973	0.2	-49
LUBRIZOL 8052	0.05	-49			
LUBRIZOL 8052	0.1	-50.5			
LUBRIZOL 8202	0.1	-42			
LUBRIZOL 8202	0.2	-43			

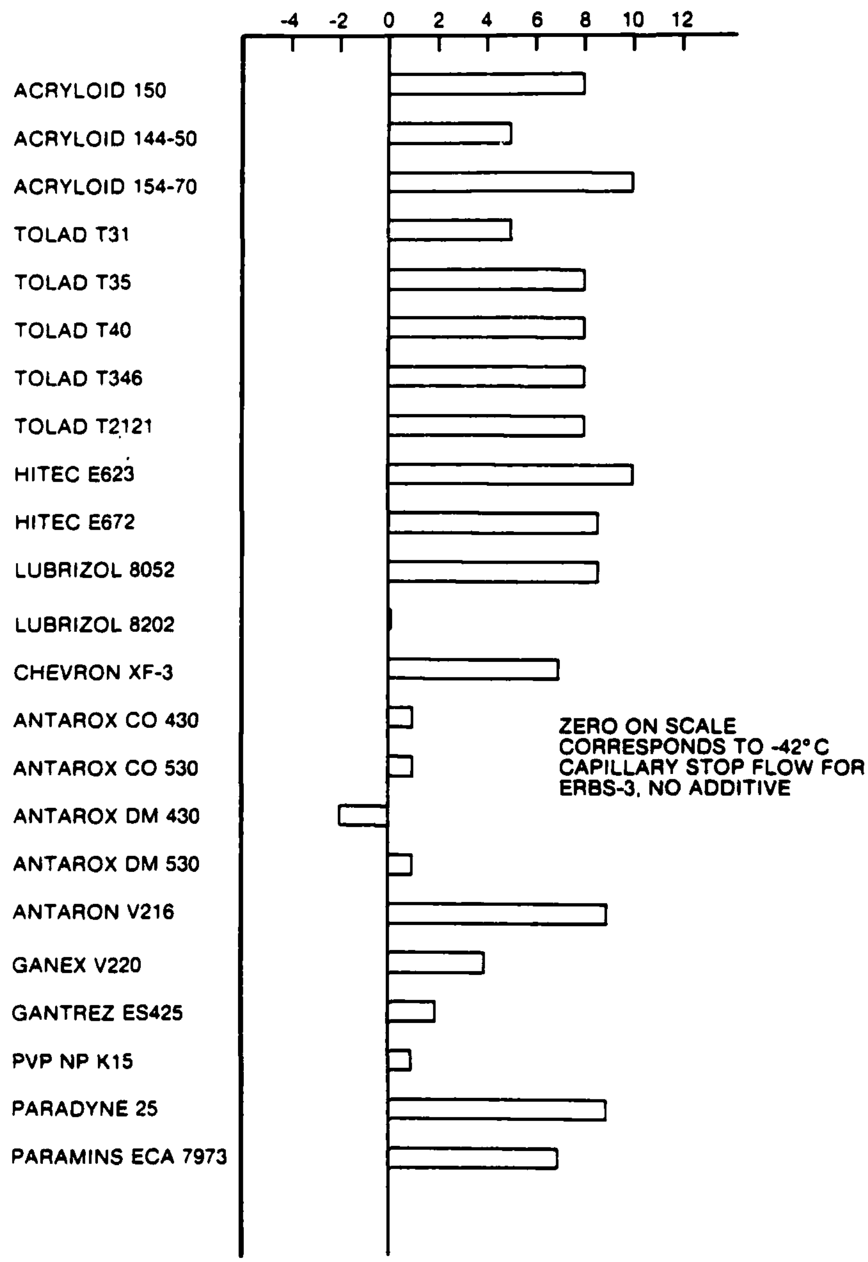

FIGURE 3. EFFECT OF 23 FLOW IMPROVERS ON CAPILLARY TEST RESULTS FOR ERBS-3

TABLE V

CAPILLARY TEST RESULTS FOR ISOOCTANE + ERBS-3
WAX DISTRIBUTION

ADDITIVE	%W/W CONC	STOP FLOW	ADDITIVE	%W/W CONC	STOP FLOW
(NONE)	-	-49	LUBRIZOL 8202	0.1	-49
ACRYLOID 150	0.1	-49	LUBRIZOL 8052	0.1	-48
ACRYLOID 154-70	0.1	-49	CHEVRON XF-3	0.1	-48
TOLAD T-31	0.1	-49	ANTARON V 216	0.1	-49
TOLAD T-35	0.1	-48.5	GANEX V 220	0.1	-49
TOLAD T-40	0.1	-48.5	PARADYNE 25	0.1	-48
TOLAD T-346	0.1	-50	PARAMINS ECA7973	0.1	-49
TOLAD T-2121	0.1	-49			
HITEC E-623	0.1	-50			
HITEC E-672	0.1	-50			

It is apparent from Figures 1 and 3 that with ERBS fuel the capillary stop flow points are depressed considerably less than the Setapoints, and also that the BP test does not discriminate well between flow improvers. Most of the additives either depressed the capillary stop point 7 - 10°C, or were completely without effect. The Antarox family again falls into the latter category. Roughly, the Acryloid, the Tolad and the Hitec series, Chevron XF-3 and Antaron V-216 are equally effective, while Lubrizol 8202 excellent in the Setapoint test, has almost no effect on the capillary stop flow. Thus there is, with this fuel, no satisfactory correlation at all between results by the two methods.

It has been observed (1) that in the tests employing ERBS, there was no particular change in response to Lubrizol 8052 as its concentration varied from 0.025 to 0.1%. This is borne out in the present work. Examining Tables II and IV, it is seen that with all the effective flow improvers the maximum depression in stop flow points is attained, or nearly so, at the lowest level (0.05%). With several of the less effective materials (e.g., Antaron V 216, Acryloid 150) there is a further reduction in Setapoint on passage from 0.1 to 0.2 % w/w. In the capillary test, however, all results with all additives are totally insensitive to concentration.

Results with the second isooctane - wax blend using the same array of flow improvers and the two test methods are gathered in Table VI and Fig. 4. The main features observed were:

a) the division of flow improvers into two distinct groups; four or five were quite effective in each test, and the remainder showed no improvement over the unadditized material.

b) disparities between the two tests, seen in their response to Acryloid 154-70, Tolad T2121 and Antaron V216.

c) the extreme depression in capillary stop flow measurements, much greater than observed elsewhere for the test, with the four flow improvers that showed any affect at all (Tolad T2121, Lubrizol 8052, Paradyne 25 and ECA 7973).

Further work with this artificial blend of waxes, not duplicating the makeup of any actual fuel, was not pursued. Building up various wax distributions in a neutral base evidently affords a way to study relations between the action of a particular flow improver and wax composition, but is beyond the scope of this survey.

TABLE VI

CAPILLARY AND SETAPOINT TEST RESULTS FOR ISOCCTANE AND C_{12} - C_{19} WAX DISTRIBUTION.
UNDERLINED VALUES DISCUSSED IN TEXT

ADDITIVE	%W/W CONC	SETA STOP FLOW	SETA RESUME FLOW	CAPILLARY STOP FLOW
(NONE)	-	-29.4	-27.6	-33
ACRYLOID 150	0.1	-30.8	-24.5	-34
ACRYLOID 144-50	0.1	-29.6	-27.1	-33
ACRYLOID 154-70	0.1	-33.8	-26.7	-35
TOLAD T-31	0.1	-29.2	-25.9	-33
TOLAD T-35	0.1	-29.8	-25.5	-36
TOLAD T 40	0.1	-29.9	-25.9	-33
TOLAD T-346	0.1	-29.9	-25.7	-33
TOLAD T-2121	0.1	-30.4	-26.5	-55
HITEC E-623	0.1	-31.1	-25.4	-35
HITEC E-672	0.1	-30.7	-25.0	-34
LUBRIZOL 8052	0.1	-38.8	-38.4	-57
LUBRIZOL 8202	0.1	-28.8	-26.0	-33
CHEVRON XF-3	0.1	-31.0	-25.5	-35
ANTAROX CO 430	0.1	-29.6	-27.7	-33
ANTAROX CO 530	0.1	-29.4	-27.4	-33
ANTAROX DM 430	0.1	-30.6	-25.5	-35
ANTAROX DM 530	0.1	-29.6	-27.3	-33
ANTARON V 216	0.1	-36.1	-27.6	-35
GANEX V 220	0.1	-30.1	-25.5	-32
GANTREZ ES 425	SAT'D	-29.6	-27.5	-33
PVP NP K15	SAT'D	-29.6	-27.7	-33
PARADYNE 25	0.1	-40.1	-38.7	-60
PARAMINS ECA7973		-34.1	-34.7	-51

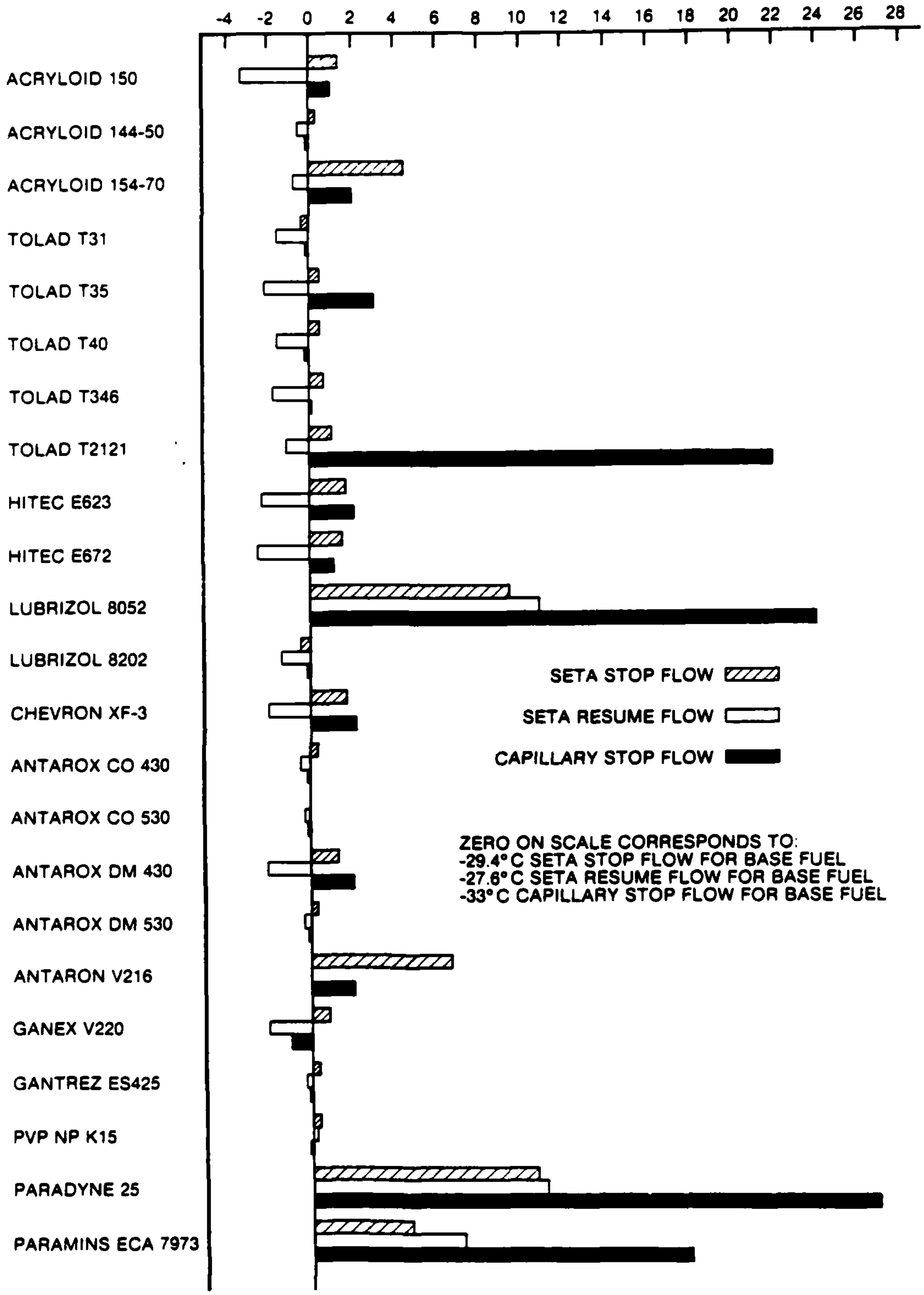

FIGURE 4. CAPILLARY TEST AND SETAPOINT RESULTS FOR ISOOCTANE WITH C_{12} - C_{19} WAX DISTRIBUTION

4.0.0 SUMMARY

1. The effect was studied, using two bench scale tests, of the addition of a number of candidate cold flow improvers, at several levels, to ERBS and to an isooctane-based synthetic fuel duplicating the wax distribution of ERBS. As ERBS is in many ways representative of what high freeze aviation fuels would be like, this gives a fair idea of the effectiveness of a number of additives, some previously untried as flow improvers, in such fuels.

2. Using the Setapoint apparatus, wax composition was the major but not the only factor in determining response to these additives.

3. The capillary test did not discriminate clearly between flow improvers added to ERBS itself, as did the Seta apparatus. Flow improver additions to the isooctane-based synthetic material had no effect on capillary test results, which were the same within experimental error as for the untreated material.

4. By both methods, the most effective flow improvers produced their maximum effect in ERBS at 0.05% w/w, further increases not depressing end points further, in agreement with previous work using this fuel.

5. Agreement between the two tests themselves was not satisfactory. Some of this is due to poor discrimination in the capillary test. There were also irregular results with certain ERBS - additive combinations in the Setapoint determination, to be discussed in a separate note.

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3. G.T. Seng, NASA Technical Memorandum 82883.

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13 ABSTRACT - Unclassified

The Setapoint apparatus, and a procedure originally developed by British Petroleum, are designed to give a measure of the minimum temperature at which middle distillate fuels can be used. These were investigated, employing an experimental fuel, ERBS, of petroleum origin, and synthetic mixtures based on isooctane with added n-paraffins. Special attention was given to the effects of cold flow improvers.

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cold flow improvers

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